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Original Article

THE CORROSION RATE OF B_4C PARTICLES REINFORCED WITH Al-Si ALLOYPREPARED BY POWDER METALLURGY IN ACIDIC SOLUTION USING RSM

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ABSTRACT

This present study is to progress the arithmetical model that might be utilized to determine the corrosion rate (CR) of Al–12Si-x B_4C (x = 0, 4, and 8 wt. % of B_4C) composites have faith in densification of the powder metallurgy throughout compaction and sintering. The acidic solutions used for corrosion is 1 N HCl, 1 N H_2SO_4 and 1 N HNO_3 for varying plummeting time (i.e., 72, 144, and 216h) correspondingly. The hardness of the composites will rise because of the increased wt. % of B_4C. The result indicates that with the enhancement of nanoB_4C particles into the matrix decrease the corrosion rate respectively. The corrosion behaviour of composites was studied by SEM and corrosion rate method. As input parameters such as reinforcement, acid and time were designed by RSM design and the response parameter was corrosion rate was obtained experimentally by the corrosion rate method. The L17 orthogonal array was selected for investigating the response surface methodology (RSM) design using three factors with one replicate. RSM design was investigated for the evaluation of interactions between response parameter and input parameters. Analysis of variance was utilized to explore the influencing input factors on corrosion rate (CR). The corrosion rate specifies varying results depending on the input values of the response parameters. The outcomes revealed that all the parameters had significant effects on the corrosion rate at 95 % confidence level.

***KEYWORDS:** Al-Si- B_4C Composites, SEM, ANOVA, RSM, Corrosion Rate*

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INTRODUCTION

Aluminium alloy reinforced with boron carbide is deliberate to be a possible substitute material with conservative monolithic aluminium alloys in numerous applications due to its enhanced specific strength and improved stiffness, less density, little thermal expansion coefficient and outstanding resistance to wear. Aluminium-based composites are broadly utilized in several industries that are not inadequate to aerospace, automotive, defense, naval, electronic packaging, thermal and sports [1]. Several techniques have been used for

manufacture aluminium-based composites, for example, liquid metal infiltration, squeeze casting, diffusion bonding, electro deposition, powder metallurgy and stir casting process. Whereas utilizing a Powder Metallurgy (P/M) method for production of aluminium based nano composites, improved mechanical properties can be achievedmeanwhilestrengthening materials are evenly disseminatedabove the matrix material [6]. Subsequently, in this methodlittle temperature is utilized for production when related to melting route, therefore it evades chemical reaction among the matrix and strengtheningmaterial [7]. An additional advantage of the P / M method is in its facility to fabricateproximate net shape component parts for the required size at little cost and offerrespectable dimensional tolerance for the difficult geometries. An economical way of solving these problems was the use of strengtheningfine particles such as B_4C, TiC, TiB_2, ZrC, SiO_2, Al_2O_3, SiC particles and whiskers or other fine particles or composites as alloyingfine particles. The inclusion of these ceramics and alloying fine particles makes it potential toenhance the specific elastic modulus of aluminium based nano composites, enrich its thermal properties etc. [2]. Bythese strengthening B_4C, the nano composites have reduced densities and are also improved wear resistance. In recent times, there has been adevelopment of interest in nano composites comprising low density and reinforcements at low cost. In general, because of a few reasons the corrosion resistance of Al composites is not exactly the Al alloys, for example, the cracks at the matrix/ strengthening interface, producing flaws, microstructural variations and galvanic possessions because of the connection of the matrix and reinforcing [4]. Some chemical treating and aerospace industries, Al composites have developed as interchange materials. Amid pickling, descaling, electrochemical scratching Al composites have been broadly utilized in numerous chemical process productions, where they much of the time interact with acids or bases, because of their wide applications. Through chemical corrosion reactions happen without separation of metal and medium and electrolytic corrosion happens if the two sections are disconnected. The advancement of the corrosion reactions might be immediate chemical or electrolytic could be in conformism with the known laws of electrochemistry and thermodynamics [5]. Hydrogen gas evolution is regularly transcendent in robust acid and alkaline solutions of metals by cathodic procedures in which corrosion is convoyed [6]. The corrosion investigations about on Al composites have demonstrated that extra pit is shaped on composites than on Al alloys. The corrosion is further damaging in acidic solution. Hydrochloric acid solution was utilized for pickling, chemical and process productions wherein Al composites are utilized [7]. In current years, aluminium alloy based metal matrix composites are being investigated as candidate materials in some applications such as automobile parts and aerospace etc., [4]. In this general RSM design was utilized for the reason that this kind of design is appropriate for products and process design, process enhancement and industrial investigation. In accumulation, after confident high-order interactions are possibly insignificant, evidence on the key effects and low-order interactions might be achieved by consecutively only a RSM design [5- 7]. Hence, this current workis an effort made to scrutinize the effort of reinforcement, acid and time input factors and arithmetical model to forecastCR of Al-12Si-x B_4C composites utilizing a Box-Behnken Design (BBD), analysis of variance, the probability and CR plot.

EXPERIMENTAL PROCEDURE

The electrolytic Aluminium and Silicon were obtained and the immaculateness of 99% and a molecule measure lesser than 20 and 40 μm from M/S. MEPCO metal powder company, thirumagalam, Tamil nadu, India. Boron carbide powder with immaculateness of 99.9% and a molecule measure lesser than 44 μm utilized as an auxiliary strengthening material was obtained from Sigma Aldrich, Germany. Nano sized B_4C particles were blended by processing B_4C micron powders with the measure of 44 μm in a ball mill utilizing a solidified tungsten carbide vial and balls with 10 *mm* in diameter. The ball to powder proportion and rotational speed were 20:1 and 300 *rpm*, correspondingly. Toluene was

utilized as a processing medium and the granulating zone involved 80% of the chamber volume. The minimum size of the elements was ≤100 *nm*, subsequently 60 *h* grinding. A SEM pictures of a specimen by examining the surface with an absorbed beam of electrons. The electrons intermingle with molecules in the specimen, generating different signals that comprise evidence approximately the specimen surface topography and combinations. VEGA3, TESCAN (Czech Republic) was used to obtain the SEM images of the specimen. The samples were first subjected to gold ion sputtering. Ion sputtering is employed to increase the conductivity of the specimen surface. The SEMmicrostructures of the composites are shown in the following figures. Figure 1 (a) demonstrates the SEM microstructure of Al elements. It can be observed that aluminium has spherical structure. Figure 1 (b) demonstrates the SEM image of Si element and it is observed to have flattened and large flake like elements.

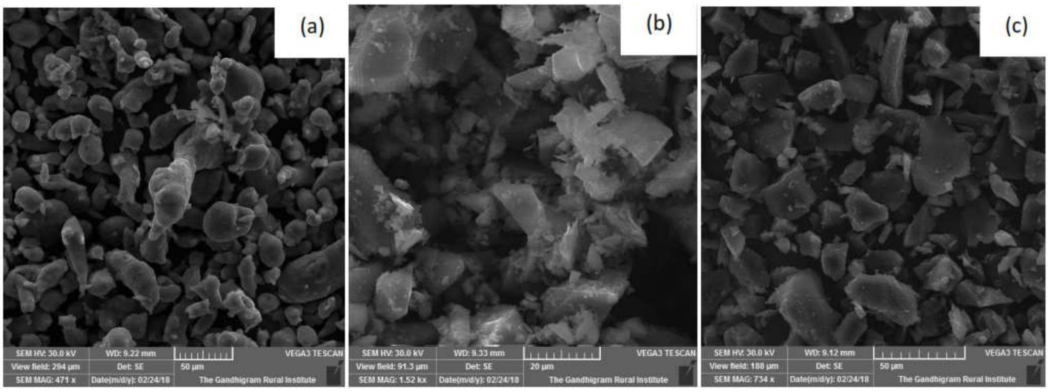

Figure 1: Demonstrates the SEM Pictures of Powders (a) Al, (b) Si and (c) B_4C

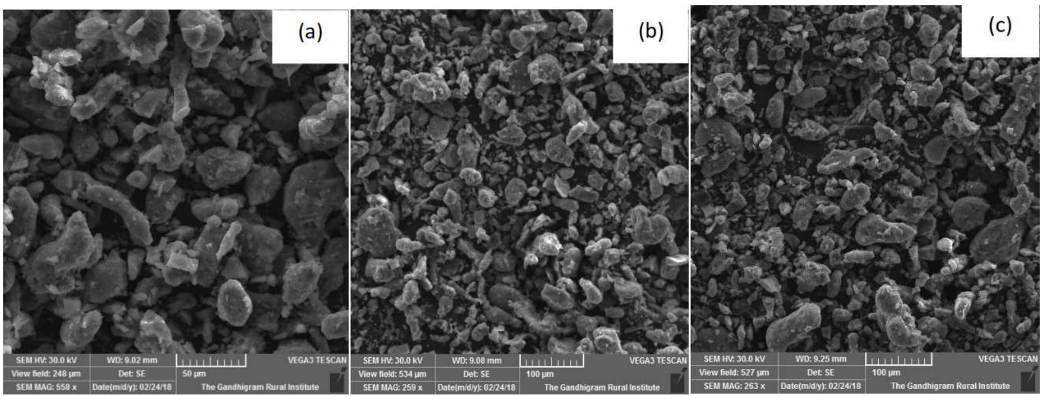

Figure 2: Demonstrates the SEM Pictures of Mixed Powders (a) Al-12Si, (b) Al-12Si-4B_4C and (c) Al-12Si-8B_4C

Figure 1 (c) demonstrates that the B_4C particles with rhombohedral shape. Rule of mixtures was utilized to evaluate the varying weight fractions of Al-12Si-xB_4C (x = 0, 4 & 8 wt.%). Mechanical Alloying (MA) was conceded out for varying combinations utilizing a ball mill with a tungsten carbide container and balls of 10 mm diameter below argon environment. The ball to powder weight ratio was 20:1 and was utilized to alloying the varying composites for each 1 *h*. To elude the rust and contamination of the MA processes, the process control agent was utilized in argon environment. From the SEM picture it was identified that the milled B_4C nanoparticle was uniformly dispersed in all the Al-12Si-xB_4C composites. The SEM picture also displays that the piercing crinkled arrangement of Al was fragmented, and for the reason that the milled B_4C particles were simply connected with the Al-12Si alloy. To rise the power of the lenient Al material irrespective of varying wt.%, milled B_4C particles were strengthenedinto the matrix. It was evidently confirmed in the

SEM pictures exposed in Figure. 2(a–c). It was perceived that all the elements were disseminated consistently through the compositions. This was owing to the stable state mingling of powders by utilizing ball mill. The alloyed powder is compacted in a compression testing machine to achieve 30 *mm*height and 10 *mm* diameter with applied pressure of 800 *MPa*. The compressed specimens are sintered using argon gas purging heating furnace for 120 *min* at 550*°C* and furnace cooled to the surrounding temperature. The sintered compressed specimen was subjected to immerse in an acidic solutions used for corrosion is 1 N HCl, 1 N H2SO4 and 1 N HNO3 for varying plummeting time (i.e., 72, 144 and 216*h*) respectively. The alloyed powder mixtures were characterized using SEM. The SEM micrograph of Al-Si, Al-12Si-4 B_4C and Al-12Si-8B_4C particles are shown in Figure 2 (a –c).

PERSUADE OF B_4C CONTENT ON DENSITY

Approximation of the Experimental Density (ED) for the sintered Al-12Si-xB_4C composites utilizing the Archimedes rule was exposed in Table 1. In Table 1 it is exposed that the ED of the sintered Al-12Si-xB_4C composites was diminished owing to the bunch of B_4C elements and openings remaining in the green specimen throughout compression. The Theoretical Densities (TD) of the composites powder blends were estimated utilizing the rule of mixtures, eliminating the sponginess. However, the ED was deliberate utilizing the Archimedes rule for a sintered specimen. The difference among the TD and ED displays the % of sponginess existing in the specimens. Consequently, the density was varying between TD and ED values. The ED of Al-12Si-xB_4C composites was improved. A weight balance with a precision of 0.001 mg was utilized to evaluate the specimens. The TD was calibrated for the Al-12Si-xB_4C composites for different composites utilizing the rule of mixtures [12]. The relative density after sintering was nearby 92% and it exposes that the sponginess of the composites has diminished by up to 8%. The wear resistance of the combinations was diminished by retaining the porosity level.

Table 1 Density of the Composites

Compositions	ED	TD
Al-12Si	2.4426±21	2.6556
Al-12Si-4B_4C	2.4283±18	2.6484
AL-12SI-8B_4C	2.4272±13	2.6412

RESPONSE SURFACE METHODOLOGY

Response surface methodology is a set of numerical and geometric methods that are helpful for budding, progressing, and bestroute. This process may be utilized by several investigators for forecasting the wt.% of composites, sliding distance, load, and so forth. It is frequently useful in conditions where the response of significance is affected by more than a few parameters and the goal is to optimize this response. The Design Expert software V11 was utilized to extend the trial design for RSM. A quadratic model of second-order form was planned to signify the connection among CR. The routine of the replica be subjected to a huge number of parameters that could be performed and relate in a difficult way. In the current work, the wt. % of B_4C, acid and time are measured as self-sufficient parameters and the response variable is the CR. In RSM, the computable form of the connection among the preferred output and the self-determining input parameters could be characterized as exposed in the subsequent equation.

$y = f (A, B, C)$

Where, y is the preferred output and fis the output function. The BBD is a first-order design improved by extra

points to permit evaluation of the modification factors of a second order model. The factorial segment of the BBD is the RSM plan with all arrangement of the factors at two levels (low -1 and high +1). The testing factors at three levels with their choice are offered in Table 2 and Table 3 exposes the trial runs with the input factors and out response.

Table 2: Experimental Factors and Number of Levels used in Design – Expert 9.0.5

Testing Parameter	Symbol	Level 1 (-1)	Level 2 (0)	Level 3 (1)
Reinforcement (A)	Wt.% of B_4C	4	6	8
Acid (B)		H_2SO_4	HCL	HNO_3
Time (C)	hr	72	144	216

Table 3: Shows the Experimental Run and Input Factors with its Response

Std Run	Run	Input Factors			Response
		WT.% OF B_4C	ACID	TIME	CR (MPY)
13	1	0	0	0	627.947
8	2	1	0	1	236.167
16	3	0	0	0	407.538
9	4	0	-1	-1	314.189
6	5	1	0	-1	758.174
12	6	0	1	1	314.189
17	7	0	0	0	191.268
2	8	1	-1	0	316.229
1	9	-1	-1	0	301.968
10	10	0	1	-1	334.919
11	11	0	-1	1	485.486
4	12	1	1	0	397.745
3	13	-1	1	0	307.51
15	14	0	0	0	314.189
7	15	-1	0	1	314.189
14	16	0	0	0	314.189
5	17	-1	0	-1	200.301

RESULTS AND DISCUSSIONS

Persuade of B_4C content on Hardness

The hardness of any material directly influences the wearresistance properties. In the present study trial experiments were first conducted to check the influence of reinforcementson microhardness. Vickers microhardness tests were carriedout at 150 gram load with dwell of 30 seconds. The results ofVickers microhardness are shown in Table 4which revealed that the presence of B_4Cparticles significantly improveshardness in comparison to Al. Further, the influence of varying weight percentages ofreinforcements is shown in Table 4, which show that hardnessincreases linearly with increase in B_4Cweight percentage. Itwas found that at higher reinforcement levels, the variation inmicrohardness occurred which may be due to theagglomeration of reinforcements in matrix phase. This aggregation of particles causes differential hardenedphases and hence causes higher variations. It can be clearlyseen that the highest microhardness of 71±14 after sintered and 76±20 VHNafter the wear testis shown by the nano composite containing $8B_4C$reinforcement. In comparison to the base alloy, the reinforced composite containing $8B_4C$ shows 19.71 and 18.42%higher microhardness. Further, to study the effect of micro hardness on tri bological properties, dry sliding weartests were performed and results

are reported in the next section.

Table 4 Shows the Vickers Hardness of the Composites

Compositions	Hardness After (VHN)
	Sintered
Al-12Si	57±0.24
Al-12Si-4B_4C	64±0.18
Al-12Si-8B_4C	71±0.14

PERSUADE OF B_4C CONTENT ON ENERGY DISPERSIVE X-RAY SPECTROSCOPY

Energy-dispersive spectroscopy is a scientific method utilized for the elemental analysis of powder samples Al, Si, B_4C, Al-12Si, Al-12Si-4B_4C, and Al-12Si-8B_4C composites respectively. It relies upon the investigation of a corresponding specific source of X-ray excitation. Its portrayal abilities were expected in extensive portion to the vital rule that every component has a sole atomic assembly permitting a sole arrangement of crests on its X-Ray range. To fortify the radiation of particular X-ray as of a composite, a high-energy ray of exciting elements, for example, electrons or protons or a beam of X-rays was centered on the sample being examined. Figure 3 (a-f) demonstrates the EDS arrangement of the distinctive composites subsequent to sintering. In this current investigation, EDS arrangement was utilized to affirm the elements existing in the Al-12Si-xB_4C composites.

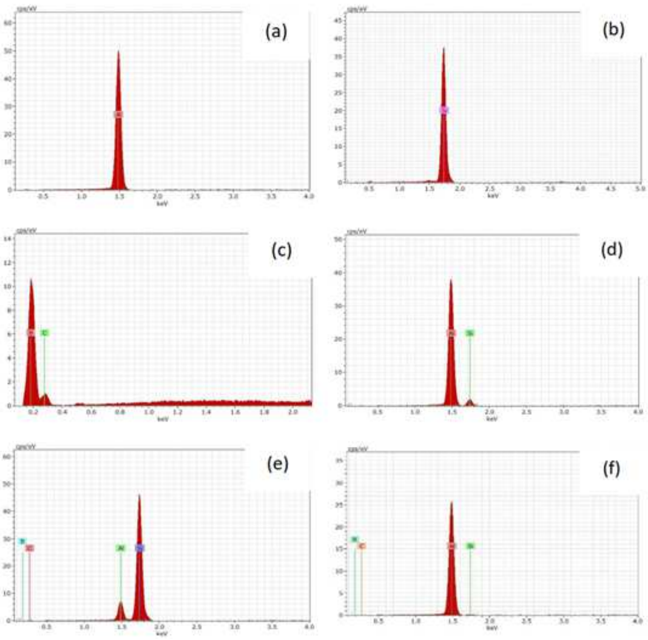

Figure 3: (a-f) Shows the EDS Graphs of the Received and Mixed Powders
(a) Al, (b) Si, (c) B_4C, (d) Al-12Si, (e) Al-12Si-4B_4C, (f) Al-12Si-8B_4C

ANALYSIS OF VARIANCE FOR CORROSION RATE

With the end goal to research the fundamentally influencing parameters the quality, ANOVA of data was executed for evaluating the impact of wt.% of B_4C, acid and time on the aggregate fluctuation of the outcomes. Furthermore, the Fisher's F-test can likewise be utilized to decide the parameters which significantly affect the response parameters when F is huge. The outcomes of ANOVA for CR of Al-12Si-xB_4C composites appear in Table 5. From the ANOVA table it is afresh explored that significance level of α=0.05, i.e. for the confidence level of 95%. It has been

adequately that P< 0.05 is implied that the conduct of the elements statistically significant or more 0.05 irrelevant to the model. Particularly the time has a more prominent impact when contrasted with the rest of the elements. Thus in this investigation, it is discovered that acid, and reinforcement elements has less influence compared to that of time on the CR separately.

Table 5:ANOVA for Corrosion Rate

Source	Sum of Squares	Df	Mean Square	F-Value	P-Value	Remarks
Model	$3.337E^{+05}$	9	37080.76	57.72	< 0.0001	significant
A-Reinforcement	50372.11	1	50372.11	78.41	< 0.0001	significant
B-Acid	65857.97	1	65857.97	102.51	< 0.0001	significant
C-Time	$1.588E^{+05}$	1	$1.588E^{+05}$	247.17	< 0.0001	significant
AB	962.51	1	962.51	1.50	0.2605	insignificant
AC	3570.17	1	3570.17	5.56	0.0505	insignificant
BC	15861.38	1	15861.38	24.69	0.0016	significant
A^2	1793.24	1	1793.24	2.79	0.1387	insignificant
B^2	4818.86	1	4818.86	7.50	0.0290	significant
C^2	31272.08	1	31272.08	48.68	0.0002	significant
Residual	4497.03	7	642.43			
Lack of Fit	4497.03	3	1499.01			
Pure Error	0.0000	4	0.0000			
Cor Total	$3.382E^{+05}$	16				

EFFECT OF INDEPENDENT PARAMETERS ON CORROSION RATE

From the consequences of the ANOVA Table 5, a model acceptability inspection was executed with a specific end goal to confirm that the quadratic model for the CR of the regression study is not abused. The normal probability curve of the residual for CR is exposed in Figure 4a which demonstrates no signal of the destruction of the independence or constant hypothesis. Meanwhile, every point in the curve takes after a straight line sequence of action, including that the errors are scattered typically. The model accomplished could be utilized to prediction the CR inside the points of confinement of the elements investigated. So as to look into the impacts of independent parameters on the CR, the two-dimensional (2D) plot and showed in Figure 4 (b-d) independently. Figures 4b and 4c show the 2D plot for theCR with the change of reinforcement × acid × time, entirely. Similarly, Figure 4d exhibits the 2D response surface plot for the CR with the change of acid × time, entirely. The results from Figures 4b and 4c show that theCR reduces with a rise in the acid, time against reinforcement. This occasion might be begun owing to the head way of a skinny carbon film at the corroded area in view of the reaction between the steel and Al-12Si-xB_4C composites in the incorporating air and moreover, the thin oxide layer can similarly go about as solid oil in this way reducing the corrosion resistance. In like manner the outcomes from Figure 4d display that the CR rises with a development in the acid and time [14]. This could be a result of the reason that with an extension in the time, the temperature augmentations to a fundamental of corrosion at which the Al-12Si-xB_4C case surface advances toward getting to be pitted. This rusty surface of Al-12Si-xB_4C whichever becomes partitioned or winds up a detectable steady to certain degree [9]. The molded separated oxide film or an element occasionally goes about as lubing and thusly this oxide film diminishes the CR. The corrosion resistance of the composites is impressively upgraded because of the consideration of the B_4C nanoparticles and ascends with expanding B_4C weight portion up to 8 wt. %. For the most part, the improved corrosion resistance of all composites is the presence of B_4C nanoparticles whose hardness is greatly improved than the matrix alloy [15]. It is notable that hard B_4C nanoparticles in the matrix alloy arrangement fortifying to the softer matrix amid acid and strengthening the Al-12Si matrix. This fortifying will confine the

deformation, and furthermore, opposes the penetration and harming of the asperities of the corrosion resistance into the surface of the composites. The B_4C nanoparticles additionally enhance stack bearing limit and warm dependability of the composites. Additionally, CR was diminished, because of the expansion in hardness of the Al-12Si-xB_4C composites. This is because of the incorporation of secondary B_4C nanoparticle fortified on the delicate Al-12Si matrix.

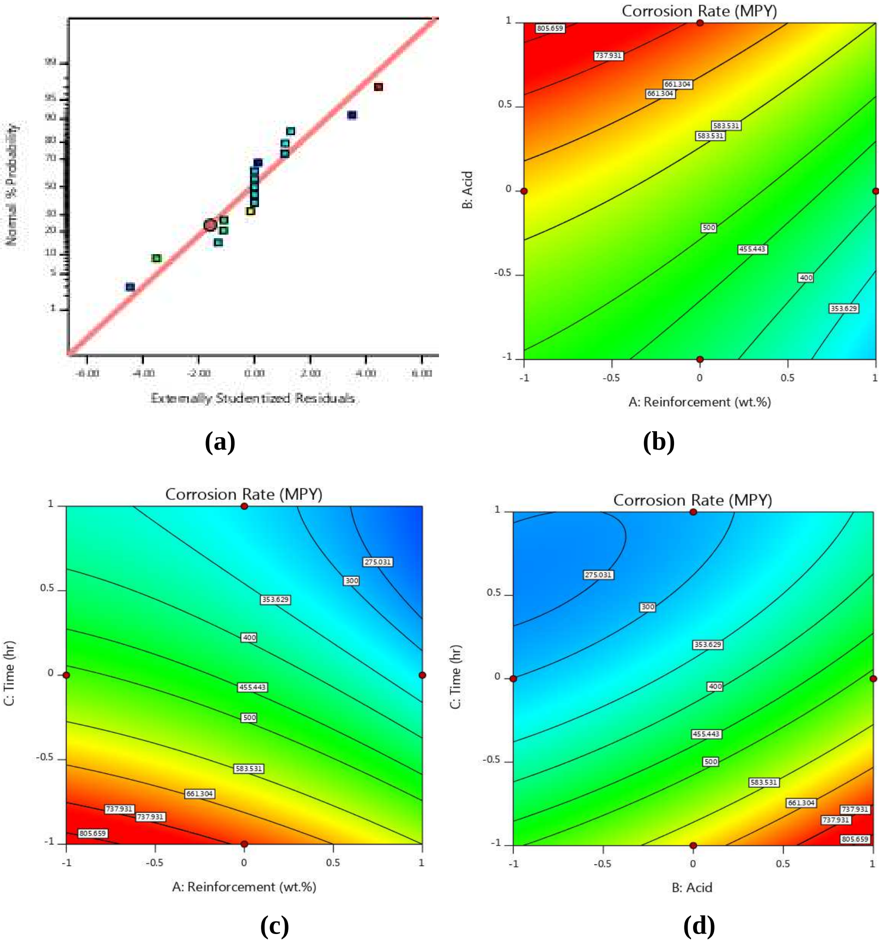

Figure 4: (a) Shows the Normal Probability Plot on Corrosion Rate and (b-d) Shows the 2D Surface Plot for Corrosion Rate of Reinforcement, Acid and Time

CONCLUSIONS

- In the present examination, the corrosion behaviour of Al-12Si-x B_4C composites were considered at room temperature with reinforcement, acid and time, by means of RSM designs to confirm the significance and effect of testing parameters on the CR.
- The hardness of the Al–12Si - xB_4C composites were expanded with expanding B_4C content support into the Al-12Si matrix.
- In view of the consequences of ANOVA, the polynomial models of the CR are all around fitted to the experimental values. The influence of the corrosion parameters on the CR was considered by the mathematical model.

- ANOVA specifies the significance of the input parameters and the % contribution of input parameters influencing on CORROSION RATE. It shows that time is the most influencing factor compared to that of reinforcement and acid.
- The experimental CORROSION RATE depends on acid, reinforcement and time and the second-order model indicated that the time had a major influence on the CORROSION RATE of the Al-12Si-x B_4C composites.
- The CORROSION RATErises with arise in thetime and acid and diminutions with arise in thereinforcement.
- The forecasted and the calibrated values areadequately near to both which specify that theimproved quadratic model could be successfully utilizedfor forecasting the CORROSION RATE of Al-12Si-x B_4C composites with 95% confidential level.

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